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RESEARCH ARTICLE

Extraction and Characterization of Nanocrystalline Cellulose from Agricultural Residue-Oil Palm Petiole Fiber

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ABSTRACT: The aim was to investigate the use of oil palm petiole fiber (OPPF) as a source for producing nanocrystalline cellulose. OPPF was treated with alkali (NaOH) first, and bleached before the production of nanocellulose by acid hydrolysis (H_2SO_4). The materials obtained after each stage of treatments were characterized and their chemical compositions were determined. The produced materials were characterized using scanning electron microscope (SEM) and Fourier transform infrared (FT-IR) spectroscopy. The morphological analysis of nanocellulose displayed clear needle like structure. The extracted cellulose has smaller diameter (8 – 10 μm) and smooth surface than the raw fibers. FT-IR results show that the hemicelluloses and lignin were entirely removed from the isolated cellulose. Fourier Transform Infrared (FT-IR) analysis showed peak at 1640cm^{-1}. This indicates that the carbonyl stretching in the acetyl groups of hemicelluloses or the ester carbonyl groups in the lignin were all removed. The results from all analysis show that OPPF is a promising good raw material that can produce of nanocrystalline cellulose.

KEYWORDS: Oil palm petiole fiber; Nanocrystalline cellulose; Acid hydrolysis.

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1. INTRODUCTION

With the exhaustion of fossil fuels and the increase in environmental problems caused by the use of fossil fuels, the development and utilization of green biomass-based materials derived from renewable natural resources have been broadly studied. Cellulose, which can be obtained in all the plant structures, is a renewable natural resource and offers the advantages of abundant availability, renewability and biodegradability [16].

Cellulose is a naturally occurring hydrophilic polymer, which forms the main part of the plant cell walls. It contributes to the physical strength of the cells [7, 9]. It has a long chain with poly (1, 4-D-anhydroglucopyranose) units that are widely spread out in wood plants (e.g., sisal cotton, flax). Cellulose can also be obtained in some marine animals and in bacteria, fungi, algae, amoeba and invertebrates [12].

The oil palm tree is a tropical plant that grows in warm climates at altitudes below 500 m above sea level. It comes from the Gulf of Guinea in West Africa, which explains its scientific name, *ElaeisguineensisJacq* and its popular name, the African oil palm tree. In Nigeria, oil palm tree is widely grown and regarded as a valuable cash crop that provides a lot of benefits including food, shelter, domestic products, employment, etc. [5,8, 11, 15]. Despite the many advantages and opportunities accruable from oil palm tree, it accounts for large amount of solid wastes at the end of its service life. The waste consists of huge amount of lingo cellulosic materials from the oil palm fronds (≈ 26.3 million tonnes), trunks (≈ 7.0 million tonnes), 23% of empty fruit bunch (EFB) per tonne of fresh fruit bunch (FFB) processed in oil

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palm mill [5,13,15]. Also, oil palm petiole is another part of oil palm tree that contains large amount of lignocellulosic biomass. It is thrown out after thinning, replanting, harvesting and pruning processes in the field resulting also to environmental problems. The enormous mass of waste obtained from the oil palm tree have led to concerns over the proper waste management system because the commonest disposal methods include; mulching, fencing, fire wood, or leaving it to degrade naturally in the plantation site. However, decomposing oil palm petiole interpose with plantation processes and creates pest breeding sites [1], especially for insects like beetles scorpions, etc. Therefore, there is need to make full use of the wastes material in the production of high value-added products, which not only complies with zero waste strategy but also generates additional profits for the farmers that grows oil palm trees. Nanocellulose is a general term for the cellulosic particles with nano-scale structural dimensions. Recently, the preparation of nanocellulose has garnered interest for being biodegradable and nontoxic, and the extraordinary properties brought by the nano-size effect [16].Nanocellulose is the cellulosic material which can be obtained from the abundance biopolymer cellulose of oil palm petiole and trunks and algae and some special bacteria [2, 9]. It is classified into three main types namely; cellulose nano-crystals (CNCs), cellulose nano-whiskers (CNWs) and bacteria cellulose (BC) [2].

Lignocellulosic fiber has gained attention and has become the major source of producing nano-cellulose due to their tremendous properties such as renewability and low cost [6, 10]. The petiole from oil palm tree is another source of lignocellulosic fibers whose utilization for the production of nano-cellulose has not been exploited. Therefore, this research opens up the opportunity or possibility of using oil palm petiole as a substitute for production of cellulose instead of using other common lignocellulosic fibers such as empty palm fruit bunch, hemp, sisal, grass and others. Hence, the aim of this study is to extract cellulose from oil palm petiole and convert it to cellulose nanocrystals (CNC) using chemical method.

2.0 EXPERIMENTAL METHODS

2.1. Materials used

Oil palm petiole cut off from oil palm tree was collected from palm plantation located at Umuagwo-Ohaji Imo State, Nigeria as raw material in this study. Reagents used include sodium hypochlorite (NaClO, 4%), glacial acetic acid, (CH_3COOH, 99.7%), sodium hydroxide (NaOH, beads, Merks), sulfuric acid (H_2SO_4), distilled water. All the chemicals are reagent grade and were used as received.

2.1.1 Preparation of oil palm petiole

Oil palm petiole (OPP) used was cut to small sizes (about 2- 3cm) and was washed thoroughly to remove dust, dirt, wax and other contaminates. This was followed by thorough drying of the samples under atmospheric air and finally ground to a size of approximately 2mm with the aid of manual grinding machine and kept in an air tight container.

2.1.2 Isolation of Nanocellulose

The dried powdered oil palm petiole sample (50g) was treated with 500 ml of 2% (w/v) sodium hydroxide (NaOH) solution for 3 h at 70^{0}C under magnetic stirring. The mixture was filtered and filtrate obtained was thoroughly washed with distilled water remove completely all the alkali residual presence on the surface. The filtrate (fibrous material) was placed in a beaker containing 450 ml of distilled water, 4% (w/v) sodium hypochlorite solution (with few drops of glacial acetic acid to adjust the pH reached 4), boiled for 8 h at 70–80^{o}C to bleach the fibrous material. The bleached fibrous material was filtered and the filtrate obtained was washed carefully with distilled water until pH value of the material gets to 7 and it was dried in air and kept in an air tight container. The essence of these alkali pretreatments were removed hemicelluloses and lignin content. The alkali treated fibrous material was hydrolyzed in 60 wt% sulfuric acid solution boiled at 45^{o}C for 30 mins under careful agitation. 10-fold of distilled water was added to quench the reaction process. The diluted suspension was centrifuged at 3000 rpm for 30 mins to obtain precipitate. The precipitate was dialyzed for 3 days using a cellulose membrane until a constant pH was reached.

2.1.3 Determination of cellulose, hemicellulose and lignin content

5wt% sodium hypochlorite solution with pH 4 was prepared by adjusting with few drops of sulpuric acid. The prepared raw fiber sample was soaked in the prepared solution for 1 h at 70 °C and the weight ratio of fiber to NaClO solution in the system is 1: 2. The mixture was filtered and the filtrate obtained (holocellulose) was washed carefully with distilled water and dried thoroughly in an oven at 60 °C. The amount of cellulose present in the fibrous material was evaluated by soaking the holocellulose for 24 h with 6 wt % of potassium hydroxide solution at room temperature. Later, the mixture was filtered and filtrate (cellulose) obtained washed with distilled water and oven-dried at 60 °C until a constant weight was obtained. The hemicellulose content of the fibrous material is the difference between the values of holocellulose and cellulose.

Furthermore, the lignin content was determined by soaking the raw fibre sample in 72 wt % H_2SO_4 acid solution for 1 h at 30 °C according to Technical Association of the Pulp and Paper Industry TAPPI standard method T222. Later, the mixture was diluted to 3% H_2SO_4 with distilled water, retained for 2 h in a reflux and filtered. The insoluble solid residue obtained was washed carefully using distilled water and oven-dried at 60 °C until a consistent weight was achieved. The similar processes were repeated for the treated fibre sample [12].

2.2. Characterization of prepared fibre samples

Both the prepared raw and treated fibre samples were characterized to determine the effect of chemical treatment on the structural and surface morphology respectively property of the fibrous materials.

(a) Structural morphology

The Fourier Transform Infrared Spectroscopy (FT-IR) spectra of raw OPP and nanocellulose fibers were obtained by using Transmittance Method spectrum One-Perkin Elmer with software spectrum version 5.02. The fibers were crushed into small particles and then blended with potassium bromide (KBr) followed by pressing the mixture into ultrathin pellets.

(b) Surface morphology

The surface morphology of the film samples were investigated by using a JEOL JSM-6390LV scanning electron microscope, with the accelerating voltage of 50 kV. All specimens were coated with gold and then observed.

3. RESULTS AND DISCUSSION

3.1. Percentage composition of fibre content

The results of percentage composition of prepared raw and chemical treated fiber samples are shown in Table 1.

Table.1: Chemical composition of raw OPPF and treated OPPF

Material	**Cellulose (wt %)**	**Hemicellulose (wt %)**	**Lignin (wt %)**
Raw OPPF	15.2	67.2	17.8
Alkaline-treated OPPF	57.8	21.2	4.5
Bleached OPPF	86.9	7.1	1.0

Data in Table 1 revealed that treatment of the prepared fibre sample with alkali (sodium hydroxide) was effective in removing surface impurities, improving the cellulose content (from 15.2 wt% to 57.8 wt % and reducing the hemicellulose, and lignin content respectively. Thus, the value of hemicellulose content of treated fibre was reduced from 67.2 to 21.2 wt %, while that of lignin content was reduced to from 17.8 to 4.5 wt %. In addition, the introduction of the fibrous material in a bleaching treatment process using sodium hypochlorite enhanced the cellulose content from 15.2 wt% to 86.9 wt % with further reduction in the value of hemicellulose and lignin content respectively. Evidently, it could deduce from Data in Table 1 that the prepared fibre sample from oil palm petiole responded positively with the treatments in removing most of the lignin and hemicellulose, resulting in high cellulose content. [12, 16].

3.1.2 Visual observation results

Figure 1 presents the results of the physical appearances or visual observation of processed raw OPPF, alkaline treated OPPF and bleached OPPF respectively. The color of the OPPF was changed as a result of the treatments. The color of alkaline-treated OPPF changed from brown to light brown which is the original color of raw OPPF, and then turned white after the bleaching process due to the removal of hemicellulose and lignin substances [3, 4,12].

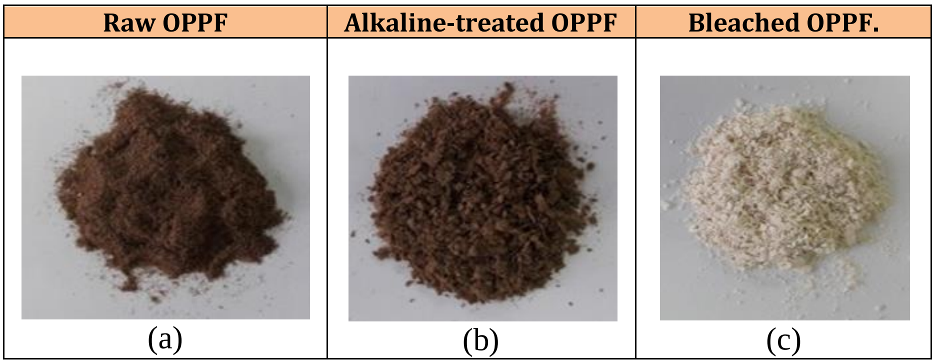

Figure.3.1: Physical display of (a) Raw OPPF, (b) alkaline-treated OPPF and (c) bleached OPPF.

3.2. Characterization Analysis

(a) Structural morphology result

This was carried out to analyze the functional groups present in OPPF internal structure and to examine the changes that took place because of the various chemical treatments. Thus, Figure 3.2 - 3.5 shows the spectra of (a) Raw OPPF (b) Alkaline treated OPPF, (c) Bleached OPPF and (d) Acid hydrolyzed OPPF (CNC).

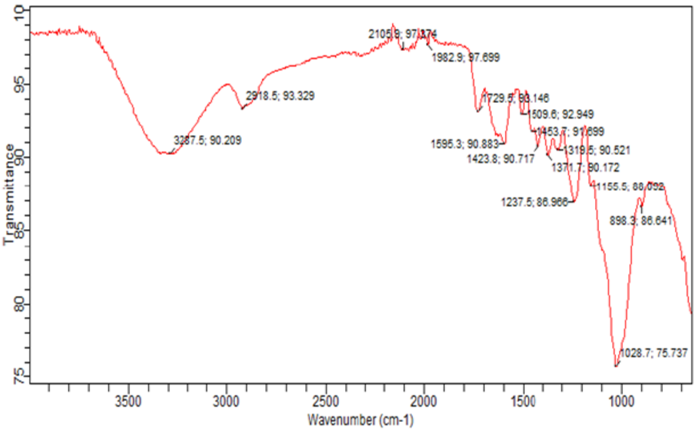

Figure 3.2: FT-IR Spectra of Raw OPPF

Since OPPF is lignocellulosic in nature, it comprises mainly of hemicellulose, lignin and celluloses. This means that the main composition of the fiber includes ketones, alkanes, and alcohols with different oxygen-containing functional groups [12].

A shoulder noticed at 1729.5 cm^{-1} for the raw OPPF is as a result of the C=O stretching vibration of the uronic ester groups and acetyl groups, from hemicellulose, pectin, or the ester linkage of carboxylic group of ferulic and *p*-coumaric acids of lignin and hemicelluloses. On the other hand, the peak at 1237.5 cm^{-1} in raw OPPF spectrum is because of the to the C–O stretching vibration of the acetyl or aryl groups in the lignin

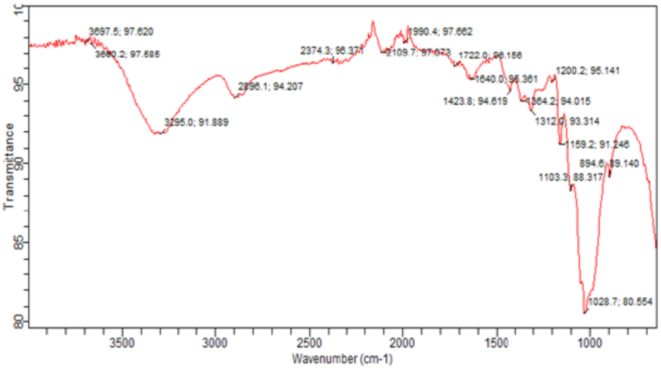

Figure 3.4: FT-IR Spectra of Bleached OPPF

The intensity of the absorbance peak at 1595.3 cm^{-1}, which reflects the C=C stretching of aromatic rings in lignin was reduced after treatments showed that the partial removal of hemicellulose, lignin, and waxy substances had occurred [13]. Meanwhile, all of the spectra exhibited around 2918.5-28850 cm^{-1} showed the characteristics of C–H stretching vibrations consist in cellulose components, interrelating to the lignin molecules. The reduction intensity at the absorption peak due to the elimination of lignin in fibers shows that the alkaline and bleaching treatment removed the lignin constituents.

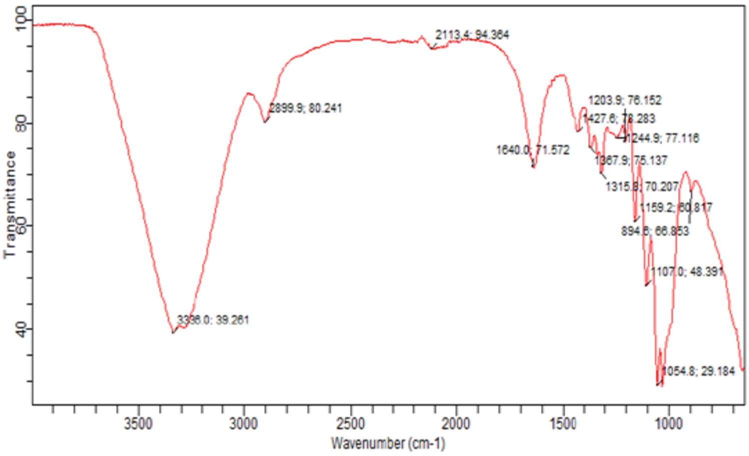

Figure 3.5: FT-IR Spectra of Acid treated OPPF

During chemical treatments of the fiber, the two peaks are gradually removed in the spectra due to the complete removal of hemicellulose and lignin during the treatments. Most researchers have all so reported the same results [12, 14].The intensity of the absorbance peak at 1595.3 cm^{-1}, which reflects the C=C stretching of aromatic rings in lignin was reduced after treatments showed that the partial removal of hemicellulose, lignin, and waxy substances had occurred [13].Meanwhile, all of the spectra exhibited around 2918.5-28850 cm^{-1} showed the characteristics of C–H stretching vibrations consist in cellulose components, interrelating to the lignin molecules. The reduction intensity at the absorption peak due to the elimination of lignin in fibers shows that the alkaline and bleaching treatment removed the lignin constituentsThe peak at 1054.8 cm^{-1} in acid hydrolysed OPPF spectrum is attributed to the S–O stretching resonance of the sulfoxide when the fibers are hydrolysed with sulfuric acid. The differences between the FTIR spectra of raw OPPF and acid hydrolysed fiber strongly demonstrated that nanocellulose was successfully isolated from OPPF by sulphuric acid hydrolysis.

(b). Surface Morphology

This was used to study the surface morphology of the OPPFs. Figure 3.6 - 3.8 shows the SEM micrographs of (a) raw OPPF, (b) alkaline/ bleached treated OPPF and (c) acid treated fiber. The several treatments performed on the fibers affected the fiber morphology, exposing its surface due to ditching of some non-cellulosic components such as pectin, hemicelluloses, lignin, wax, and other impurities from the fiber causing peeling off of nanofibrils bundles from fiber surface. In Figure 3.6, raw OPPF consists of bundles of unbroken single cells bonded together by cement materials. The surface of raw OPPF was non-uniform, rough, and caked with some contaminants such as hemicellulose, lignin, pectin, and waxy elements. After the alkaline/ bleaching treatment, the surface morphology of OPPF appears relatively clean and porous as can be observed in Figure 3.7. The pretreatments of the fiber by alkaline and bleaching process have destroyed the lignocellulosic complex, solubilized the lignin and hemicellulose to uncover the surface area of the hidden cellulose. This resulted in the cellulosic fibers aligning individually between each other, making them more available for the cellulose nanocrystal extraction process. [11,13].Colour and morphological changes were due to the removal of lignin and other extractives during alkali and bleaching treatments, resulting in lighter colour and uniform surface of the fibers, respectively. The extracted cellulose fibers had smaller diameters (8 – 10 µm) and smoother surfaces compared to the raw fibers, which had diameters ranging from 150 to 300 µm and non-uniform surfaces [11, 13].

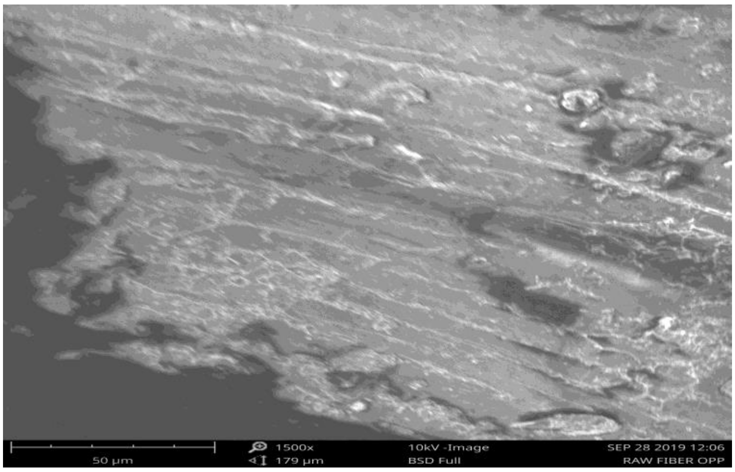

Figure 3.6: SEM Results of Processed Raw OPPF

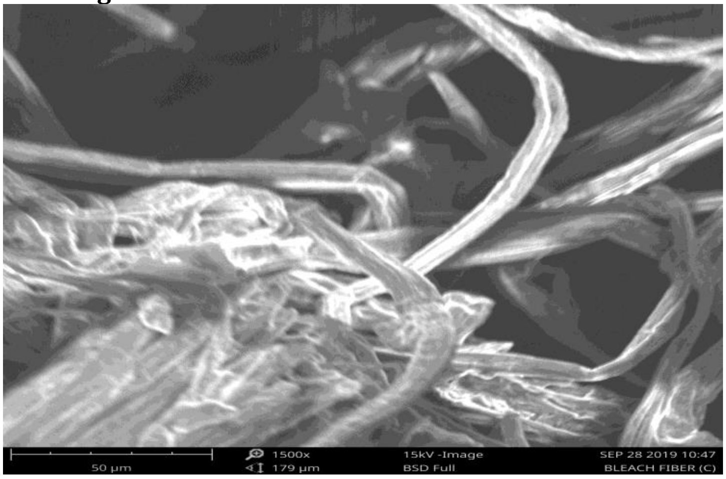

Figure 3.7: SEM Result of Bleached OPPF

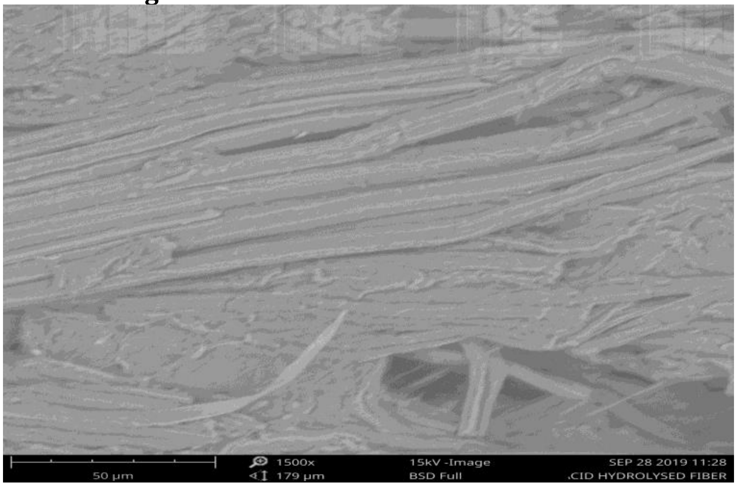

Figure 3.8: SEM Result of Acid Treated OPPF

CONCLUSION

The extraction of CNC from OPPF was successfully performed by chemical treatment through alkali, bleaching and sulphuric acid hydrolysis. The physicochemical characterization of CNC extracted from OPPF showed high level of purity and high crystallinity. FTIR analysis showed that the lignin and hemicellulose of OPPF were successfully removed after the bleaching process. SEM images showed remarkable changes on the surface morphologies of the fibers. The fiber surfaces became smoother and finally reduced in diameter and size after acid hydrolysis. The isolated nanocrystalline cellulose displayed their rod-like shape which further established the size reduction of OPPF to a nanometer scale of 1–6 nm in diameter. The obtained results suggest that OPPF have the potentials of being a source of fiber for the production of CNCs which can be used in pharmaceutical and polymeric applications. The successful utilization of OPPF to produce nanocellulose will create jobs and increase the economy of the country.

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